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## **Kinetics of Pressure-Induced $\alpha \rightarrow \omega$ Transformation in Titanium**

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### **Abstract**

The  $\alpha$ -phase (hcp) of titanium, which exists at normal temperature and pressure, transforms to a simple hexagonal structure ( $\omega$ -phase) at high pressure. The kinetics data obtained earlier under isothermal-isobaric conditions were found to satisfy an equation of the type  $\zeta = 1 - \exp(-t/\tau)^n$ , where  $\zeta$  is the volume fraction of the transformed  $\omega$ -phase after a time  $t$ , and  $\tau$  and  $n$  are constants at a given pressure. The characteristic time  $\tau$  decreases rapidly with increasing pressure, indicating that the kinetics of transformation increases exponentially as the pressure is increased. The pressure-dependence of  $\tau$  was used to predict the transformation pressure under shock loading. In this article, the earlier kinetics data have been re-interpreted to discuss the occurrence of  $\alpha \rightarrow \omega$  transformation in titanium under shock loading.

### **1 Introduction**

The crystal structure of pure titanium at normal temperature and pressure is hexagonal close-packed ( $\alpha$ -phase), which transforms<sup>1</sup> to a simple hexagonal structure ( $\omega$ -phase) at high pressure. The early experiments<sup>2,3</sup> have shown that the pressure induced  $\alpha \rightarrow \omega$  transformation is time-dependent. Later, careful experiments<sup>4,5</sup> were done to obtain the kinetics data at various pressures under isobaric-isothermal conditions. A model which involves a pressure-dependent activation energy barrier was used to interpret the kinetics data. A detailed discussion of the kinetics of pressure-induced phase transformation can be found in earlier articles<sup>6-8</sup>. In this article, the kinetics data obtained earlier<sup>4,5</sup> have been re-interpreted. The occurrence of  $\alpha \rightarrow \omega$  transformation under shock loading has been examined in the light of this re-interpretation.

## 2 Background information

### 2.1 Static pressure data

In this section, only those aspects of the transformation in Ti, which are relevant to the present discussion, are given. The details of the various other aspects of this transformation can be found in a review article<sup>9</sup>.

The pressure-temperature phase diagram of titanium is shown in Fig 1. The  $\alpha \rightarrow \omega$  phase boundary is the locus of the start pressure,  $p_s$ , at different temperatures, the start pressure being the pressure at which the transformation begins when the pressure is continuously increased. Similar interpretation holds for the  $\omega \rightarrow \alpha$  phase boundary. The transformation exhibits a large hysteresis, to the extent that the  $\omega$ -phase is retained at room temperature when the specimen in  $\omega$ -phase under pressure is depressurized to one atmosphere<sup>10</sup>. The retained  $\omega$ -phase transforms back to the  $\alpha$ -phase on heating the specimen to  $\sim 400$  K. The  $\omega$ -phase was found to be stable under prolonged heating up to 373 K. The traces of the  $\alpha$ -phase were noticed in the X-ray diffraction patterns only when the specimen in  $\omega$ -phase was heated at 394 K for 8 h. This indicates the time-dependent nature of the transformation. The  $\omega \rightarrow \alpha$  transformation temperature will be used in this article to discuss the presence of  $\omega$ -phase in the specimen recovered after the shock compression. Since the time duration of the shock compression is a few  $\mu\text{s}$ , and the shock residual temperature is retained only for a short duration ( $\sim \text{min}$ ), the  $\omega$ -phase is likely to be retained up to much higher temperatures than 380 K found in the literature.

The thermodynamic equilibrium pressure  $p_o(T)$  at a temperature  $T$  can be obtained by taking the average of the start pressures of  $\alpha \rightleftharpoons \omega$  transformations at a temperature  $T$ . A line through the equilibrium pressure points gives the equilibrium  $\alpha \rightleftharpoons \omega$  phase boundary. Since,  $\omega \rightarrow \alpha$  transformation is not observed below  $\sim 400$  K, the equilibrium phase boundary constructed in this manner is limited down to 400 K. An extension of this boundary to room temperature gives  $p_o = 2$  GPa. The hysteresis of  $\alpha \rightleftharpoons \omega$  transformation decreases considerably in the presence of shear stresses. This fact was made use of in getting an independent estimate<sup>2</sup> of  $p_o$  at room temperature. The specimen was pressed between a pair of anvils, and the shear deformation was introduced by rotating one of the anvils through an angle of  $15^\circ$ . The  $\alpha \rightarrow \omega$  and  $\omega \rightarrow \alpha$  transformations were identified by breaks in the torque (required to rotate the anvil) versus pressure curves. It is seen that  $p_s$  for  $\alpha \rightarrow \omega$  transformation decreases

while  $p_s$  for  $\omega \rightarrow \alpha$  transformation increases; thus the hysteresis is considerably reduced. These experiments gave  $p_o = 2 \pm 0.3$  GPa at room temperature. A good agreement between the  $p_o$  values obtained by the extension to the room temperature of the high temperature equilibrium phase boundary and from the shear experiments indicates that  $p_o$  is not affected by the presence of shear stresses.

The start pressure of the  $\alpha \rightarrow \omega$  transformation depends strongly<sup>2</sup> on the impurities present in the specimen. In general, the impurities tend to raise the start pressures. Because of its high affinity for oxygen, Ti contains oxygen as one of the impurity elements. In the experiments<sup>3</sup> with Ti specimens containing different oxygen content, the transformation was found to start (with 3-5 min waiting period) at 6.0 GPa for specimens with  $\sim 1000$  ppm oxygen, whereas the transformation was not observed up to 8 GPa in the specimen containing 3800 ppm oxygen. The large differences in the start pressures reported by the various investigators are because of the varying level of impurities (mainly oxygen) present in the specimens used by them. This factor should be kept in mind while attempting a quantitative intercomparison of the data from different sources.

## 2.2 Shock pressure data

The fact that the  $\omega$ -phase is retained at one atmosphere on release of pressure suggested the possibility of detecting the  $\omega$ -phase in the Ti specimen recovered after shock compression. However, since the duration of the high pressure pulse in shock wave experiments is  $\sim 1 \mu s$ , the time scale of transformation should be of this order for the transformation to occur under shock compression. As discussed later in this article, the pressure dependence of the kinetics of  $\alpha \rightarrow \omega$  transformation measured in static pressure experiments suggests that the time of transformation can indeed become of the order of a  $\mu s$ . Further, the shock compression results in the heating of the specimen. In order to prevent the  $\omega \rightarrow \alpha$  transformation, the shock residual temperature should be below 400 K.

The first shock recovery experiments to detect the  $\omega$ -phase on Ti were performed by Kutsar *et al*<sup>11</sup>. The iodide grade Ti specimen at 293 K was shock-compressed to 35 GPa. The technique employed shock-compressed the specimen not in a single shock but by employing multiple reflection of waves from the walls of the specimen container. The recovered specimen contained only a small fraction of the  $\omega$ -phase. When the specimen was precooled to 120 K and shock compressed to 35 GPa, 60-70

%  $\omega$ -phase was detected in the recovered specimen. The reason for this became clear when the shock residual temperature was estimated<sup>11</sup> (Fig 2). The estimated residual temperature was above 400 K for initial specimen temperature of 293 K; this caused the  $\omega$ -phase produced during shock compression to transform back to the  $\alpha$ -phase. On precooling the specimen, the shock residual temperature was well below 400 K; this facilitated the retention of the  $\omega$ -phase in the shock compressed specimen. It may be noted that at high enough pressure (above 60 GPa), shock residual temperature may become so large as to reach the  $pT$ -region where  $\beta$ -phase is stable.

Fig.1

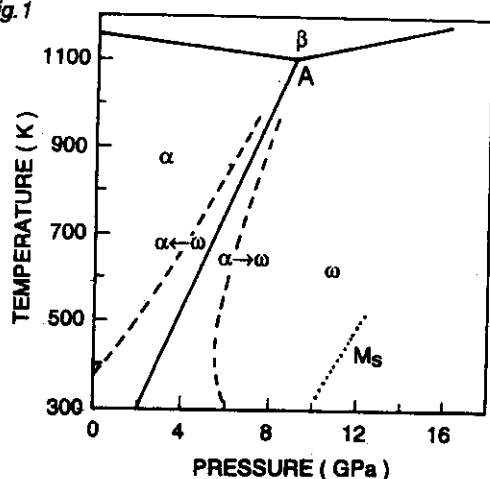


Fig.2

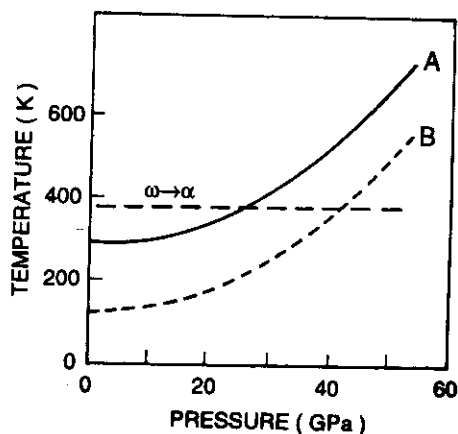


Figure 1: The phase diagram of Ti(ref 2). The triple point (A) occurs at  $p=9$  GPa and  $T=1100$ K.  $\alpha$  - hcp,  $\beta$  - bcc, and  $\omega$  - simple hexagonal.

Figure 2: The estimated shock residual temperature in Ti. A - for specimen shock compressed at 293 K (ref 11). The shock residual temperature (curve-B) for specimen compressed at 120 K was obtained (our estimate) by shifting down curve-A by 173 K along the temperature axis. The broken line marks  $\omega \rightarrow \alpha$  transformation temperature at one atmosphere.

In a more detailed study, Kutsar and German<sup>12</sup> shock compressed under multiple shock condition the Ti specimen to 12, 20, 30 and 50 GPa, and examined the recovered specimens for the presence of  $\omega$ -phase. No  $\omega$ -phase was detected after shock compression at 290 K. With the specimen precooled to 120 K, the fraction of

$\omega$ -phase progressively increased from 6% at 12 GPa to 54 % at 50 GPa. The experiments involving single wave shock compression of the specimen at 120 K were conducted at 9, 14, 26.5 and 38.6 GPa. Traces of the  $\omega$ -phase were detected in the specimen compressed to 9 GPa. The fraction increased to 25 % at 14 GPa. No  $\omega$ -phase was detected in the specimen compressed at 26.5 and 38.6 GPa. The shock residual temperature after a single wave shock compression appears to be higher than if the same pressure is reached in multiple wave shock compression.

It may be noted that even for the specimen cooled to 120 K, the estimated shock residual temperature is  $\sim 400$  K for a compression to 40 GPa, and well above 400 K for 50 GPa (Fig 2). The fact that the fraction of retained  $\omega$ -phase was largest for 50 GPa compression suggests that some other factors in addition to the shock residual temperature must be considered while discussing the presence of  $\omega$ -phase in shock-recovered specimens.

The profiles of the shock waves in Ti were measured<sup>13</sup> at a number of pressures in the range 5.8 and 24.9 GPa, presumably at room temperature. The splitting of the wave was observed at 14 GPa. This was indicative of a first-order transformation (presumably  $\alpha \rightarrow \omega$ ), and the transformation pressure (equal to amplitude of the shock wave) was 11.9 GPa. However, no splitting of the wave was observed between 17.7 and 24.9 GPa, indicating the absence of any phase transition. The absence of  $\omega$ -phase in the specimen recovered after the shock compression (with the specimen initially at room temperature) is understandable, as the shock residual temperature is higher than the  $\omega \rightarrow \alpha$  transformation temperature at one atmosphere. On shock compression to the pressures ranging between 17 and 25 GPa, the specimen is well within the region of stability of  $\omega$ -phase, and therefore, the absence of the  $\alpha \rightarrow \omega$  phase transformation in the pressure range 17.7 to 24.9 GPa remains unexplained.

The work of McQueen *et al*<sup>14</sup> is the earliest on shock compression of Ti. These authors reported a small discontinuity at 17.5 GPa in the particle-velocity versus wave-velocity data, and assumed this feature to be associated with the  $\alpha \rightarrow \beta$  transformation. It is stated<sup>14</sup> that "the assumption was strengthened but not verified by the fact that the shock-loaded Ti was recovered in the bcc phase" The volume change associated with this discontinuity is 1.1 % which agrees well with the volume change of 1.5 % observed for  $\alpha \rightarrow \omega$  transformation. This prompted Kutsar and German<sup>12</sup> to state that "the phase transition detected (by McQueen *et al*<sup>14</sup>) should be correlated

with  $\alpha \rightarrow \omega$  transition but not  $\alpha \rightarrow \beta$  one". However, this issue re-surfaced with added confusion after Kutsar *et al*<sup>13</sup> failed to detect the splitting of the shock wave profile in the pressure range of 17.7 to 24.9 GPa.

### 2.3 Nature of transformation

The  $\alpha \rightarrow \omega$  transformation is martensitic, exhibiting well defined orientation relations<sup>15,16</sup>. The kinetics under static compression is found to be sluggish and time-dependent. (A detailed discussion of the kinetics aspect is given in the next section). The time-dependence of the transformation is reminiscent of the isothermal martensitic transformations<sup>17,18</sup> occurring at constant (low) temperature, and analogously the pressure-induced time-dependent transformations are called isothermal-isobaric. In such pressure-induced transformations, nucleation of the high pressure phase faces an activation energy barrier which is overcome basically by thermal fluctuations. Once a critical size of nucleus is formed, transformation proceeds spontaneously. Thus, the observed kinetics is largely the kinetics of nucleation. As discussed in the next section, the activation barrier decreases rapidly with increasing pressure. Vohra<sup>19</sup> has discussed various other models which can explain the time-dependence of the transformation.

The  $\alpha \rightarrow \omega$  transformation in Ti also takes place under shock loading. Since the duration of the shock pressure-pulse is small, it is conjectured that if a transformation occurs under shock loading, then it must occur more like an athermal martensitic transformation. The pressure-induced transformations of this type, called athermal-isobaric, are thought of occurring spontaneously without nucleation<sup>20</sup> under the driving force provided by the over-pressure. We do not propose debating whether the athermal martensitic transformation occurs with or without nucleation, but simply note here that these considerations suggest a change in the nature of transformation from isothermal-isobaric under static pressures to athermal-isobaric under shock loading; this transition line is marked M<sub>s</sub> in Fig.1.

## 3 Kinetics under static pressures

The  $\alpha \rightarrow \omega$  transformation in Ti results in a large increase ( $\sim 20\%$ ) in the electrical resistance of the specimen (Fig 3). This feature was used to obtain the fraction of the transformed  $\omega$ - phase,  $\zeta$ , as a function of time,  $t$ , at constant pressures ranging from 4 to 9 GPa and temperature  $300 \pm 3$  K. A tungsten carbide opposed anvil setup with pyrophyllite gasket and talc pressure transmitting medium was used to

pressurize the specimen. The experiments were carefully designed to achieve isobaric-isothermal conditions. Since, the thermodynamic equilibrium pressure,  $p_o$ , is 2 GPa, the over-pressure employed in these studies ranged from 2 to 7 GPa. The thermal activation aspect of the transformation was examined by measuring the kinetics at constant pressure and elevated temperatures. The details of this work can be found in earlier articles<sup>4,5,21</sup>. The following features of the kinetics data were observed.

### 3.1 General features

The transformation is preceded by an incubation period which decreases exponentially as the pressure is increased. Typically, the incubation time is  $10^5$ s at 4 GPa and decreases to  $10^2$ s at 6 GPa. The transformation runs to completion as the pressure is held constant. The  $\zeta$ - $t$  data at two different pressures are shown in Fig 4.

The  $\zeta$ - $t$  data satisfy an equation of the form,

$$\zeta = 1 - \exp - (t/\tau)^n \quad (1)$$

in the entire pressure range (Fig 5). At a constant pressure, both  $\tau$  and  $n$  are constant terms. With increasing pressure,  $\tau$  decreases rapidly, indicating that the kinetics becomes faster as the pressure is increased. The  $\ln\tau$ - $p$  data, (Fig 6), exhibit a small curvature at 6 GPa. Since the transformation does not occur at  $p = p_o$ ,  $\tau$  should asymptotically approach infinity as  $p = p_o$  is approached. On fitting through the  $\ln\tau$ - $p$  data an equation of the form,

$$\ln\tau = a_1 + a_2(p - p_o) + a_3(p - p_o)^{-2} \quad (2)$$

the following values were obtained<sup>5</sup>:  $a_1 = 13.36$ ,  $a_2 = -1.51$ ,  $a_3 = 23.7$  and  $p_o = 1.5$  GPa. This analysis is of physical significance as it gives a value of  $p_o$  which is in reasonable agreement with the value obtained by other methods.

### 3.2 Energetics of transformation

Assuming thermally activated nucleation and growth processes, it can be easily shown<sup>22</sup> that the time  $t_\zeta$  required for a fraction  $\zeta$  is given by,

$$\ln t_\zeta = b(\zeta) + \Delta E(p)/kT = b(\zeta) - (\Delta S/k) + \Delta H(p)/kT \quad (3)$$

where  $b(\zeta)$  is a term which depends only on  $\zeta$ , and  $\Delta E(p)$  and  $\Delta H(p)$  are the pressure-dependent activation free energy and enthalpy respectively for the combined processes

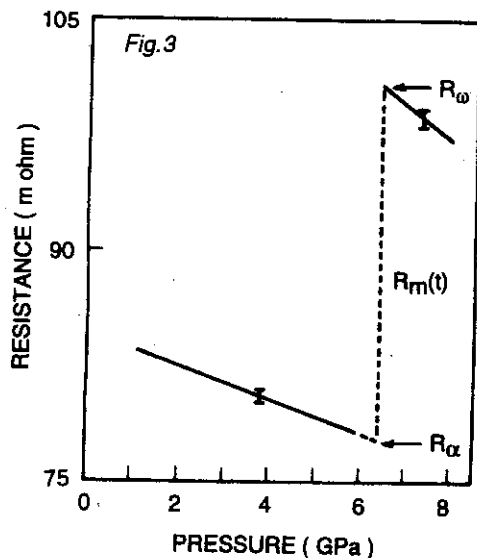


Figure 3: Variation of the resistance of Ti with pressure in a typical run; the jump in resistance indicates the  $\alpha \rightarrow \omega$  transformation.  $R_\alpha$  and  $R_\omega$  denote the specimen resistance in fully  $\alpha$ -phase and  $\omega$ -phase respectively.  $R_m(t)$  is time-dependent resistance of the specimen undergoing  $\alpha \rightarrow \omega$  transformation.

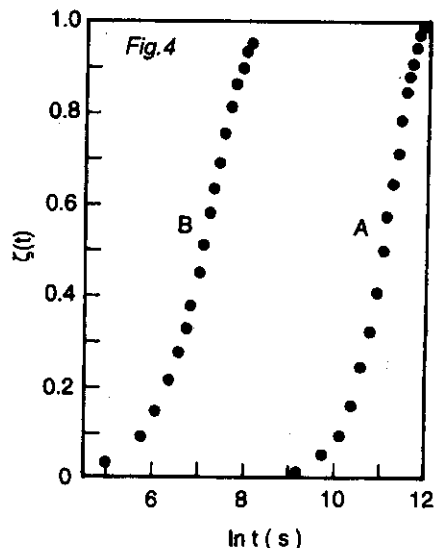


Figure 4: Typical  $\zeta$  versus  $t$  data for Ti at two different pressures. A - 4.5 GPa; B - 6.2 GPa.

of nucleation and growth. The entropy term is assumed to be independent of pressure. The contribution from the entropy term is estimated to be  $\sim 1$  kcal mole $^{-1}$ . The measurement<sup>5</sup> of the temperature-dependence of  $\tau$  at different pressures showed that at each pressure,  $\ln \tau$  indeed varied linearly with  $(1/T)$ . These data gave  $b(0.63) = -14 \pm 1.5$ . In view of a large error, which is of the order of the contribution from the entropy term, we drop the distinction between  $\Delta H(p)$  and  $\Delta E(p)$ , and retain  $\Delta E(p)$  in the following discussion. Since  $\ln(10^{-6}) = -13.81$  and the measured value of  $b(0.63)$  is  $-14 \pm 1.5$ , the value of  $\Delta E(p)$  corresponding to  $\tau = 1 \mu\text{s}$  is  $0.2 kT$ . Since there is a large inherent uncertainty in the measurement of  $b(0.63)$ , a value of  $-13.81$  has been taken for  $b(0.63)$  instead of  $-14$ . With  $b(0.63) = -13.81$ ,  $\tau = 1 \mu\text{s}$  will occur when  $\Delta E(p) = 0$ . On combining Eqs(2) and (3), the following expression for the



activation free energy is obtained,

$$[\Delta E(p)/kT] = 27.17 - 1.51(p - p_o) + 23.7(p - p_o)^{-2} \quad (4)$$

The  $\Delta E(p)$  versus  $p$  curve represented by Eq(4) is shown in Fig 7. These data suggest that the  $\alpha \rightarrow \omega$  transformation in Ti is thermally activated, and the kinetics becoming faster is simply a consequence of the activation free energy decreasing with increasing pressure.

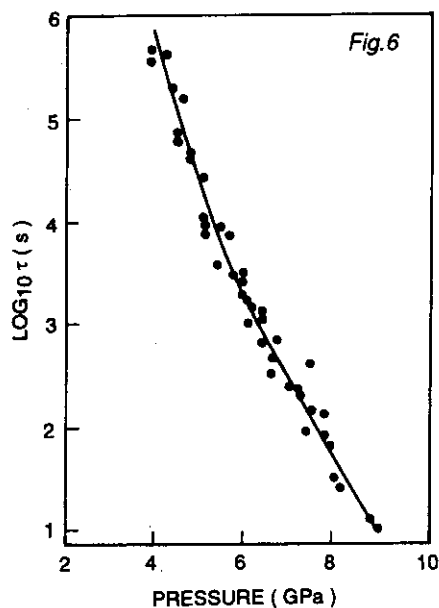
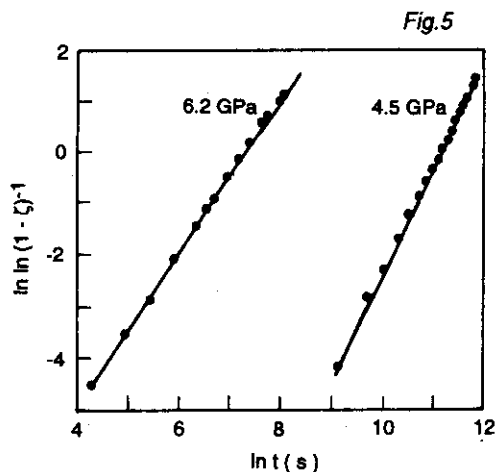


Figure 5: The  $\ln \ln(1 - \zeta)^{-1}$  versus  $\ln t$  plots are linear indicating that the  $\zeta - t$  data fit an equation of the form  $\zeta = 1 - \exp - (t/\tau)^n$  where both  $\tau$  and  $n$  are constants at a given pressure.

Figure 6: Pressure dependence of  $\tau$  at 300K. It may be noted that at  $t = \tau$ ,  $\zeta = 1 - \exp(-1) \simeq 0.63$ .

It is seen from Eq(3) that

$$\tau = \tau_o \exp[\Delta E(p)/kT] \quad (5)$$

where  $\ln \tau_o = b(0.63)$ .

Thus,  $\tau_o$  is the minimum value of  $\tau$  which occurs at  $\Delta E(p) = 0$ . The limited experimental data<sup>3</sup> suggest that the kinetics of  $\alpha \rightarrow \omega$  transformation in Ti also depends on the impurities present in the specimen and the grain size. Both  $\Delta E(p)$  and  $\tau_o$ , therefore, should be considered functions of the impurities and grain size. If it is assumed that these factors affect only  $\Delta E(p)$  and its pressure dependence, then  $\tau_o$  will emerge as a true material constant characterizing the transformation. Since  $\tau_o$  is the measure of time for the propagation of the transforming phase in absence of any energy barrier, it is expected to depend only on the elastic properties of the specimen material.

### 3.3 Pressure-dependence of $n$

The kinetics data<sup>4,5</sup> suggest that  $n$  in Eq.(1) is pressure-dependent. Though the  $\ln \ln(1-\zeta)^{-1}$  versus  $\ln \tau$  plot in each individual run results in a good straight line as shown in Fig 5, the  $n$ -values obtained from these data, however, exhibit a large scatter in the  $n$ - $p$  plot (Fig 8). This scatter appears to be inherent in this type of measurements. The following form for the pressure-dependence of  $n$  is suggested by the  $n$ - $p$  data in the pressure range 4 to 9 GPa,

$$n = 1.52 + 0.26p - 0.035p^2 \quad (6)$$

If it is assumed that the  $n$ - $p$  relation predicted by Eq.(6) does not change drastically in the vicinity of  $p > 9$  GPa, then it is seen from Eq.(6) that  $n = 0$  at  $p = 11.4 \pm 0.4$  GPa.

## 4 Transformation under shock loading

The duration of the pressure pulse in shock loading is  $\sim 1 \mu\text{s}$ . A transformation can occur under such a condition only if the  $t_c$  can be  $\sim 1 \mu\text{s}$ . Since  $\tau$  (and in general  $t_c$ ) is experimentally found to decrease with increasing pressure, it appears possible that at high enough pressures  $\tau \sim 1 \mu\text{s}$ . The extrapolation of the  $\ln \tau$ - $p$  data to  $\tau = 1 \mu\text{s}$  gave a pressure of 19 GPa which was suggested<sup>4</sup> as the transformation pressure under shock loading. Since the shock compression is accompanied by a large shear component and rise in temperature, the kinetics of transformation under shock loading is expected to be faster than that predicted by the extrapolation of the static pressure data. Thus, the kinetics of  $\alpha \rightarrow \omega$  transformation in Ti, which is thermally

activated and exhibits the characteristics of isothermal-isobaric transformation, can become fast enough (at high pressure) to respond to short duration shock wave pressure pulses. This does not call for a change in the nature of transformation from isothermal-isobaric to athermal-abaric. However, an extrapolation of the  $\ln \tau$ - $p$  data to  $p = 19$  GPa must be interrupted (and possibly invalidated) by  $n$  vanishing at

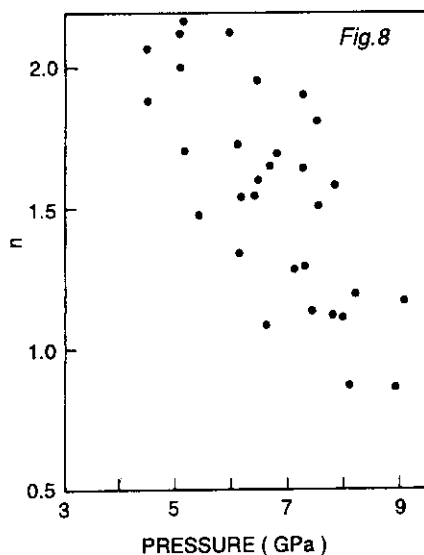
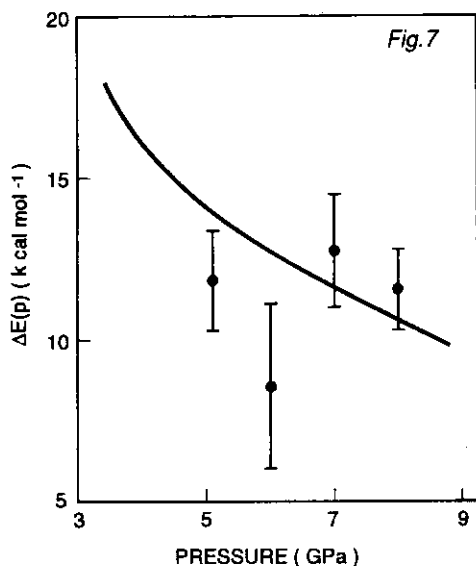


Figure 7: The solid line shows the pressure dependence of  $\Delta E(p)$  as obtained from Eq.4. The  $\Delta E(p)$  obtained from the slope of  $\ln \tau$  versus  $1000/T$  plots are shown by the dots.

Figure 8: Pressure dependence of  $n$  for the  $\alpha \rightarrow \omega$  transformation in Ti.

11.4 GPa. It is seen from Eq.(1) that with  $n = 0$ , the transformation becomes time-independent. The pressure,  $p_m$ , at which  $n = 0$ , appears to mark the transition from isothermal-isobaric to athermal-abaric character of the transformation. The study of the kinetics data at  $p > 9$  GPa will indicate whether this transition takes place continuously over a pressure range or discontinuously at  $p_m$ . The present estimate of  $p_m$  ( $= 11.4$  GPa) lies close to the start line of athermal-abaric  $\alpha \rightarrow \omega$  transformation ( $M_s \sim 10$  GPa) suggested by the shock wave data<sup>12</sup>

## 5 Some comments on JMA equation

The equations describing the kinetics of phase transformations were derived<sup>23-26</sup> based on a nucleation and growth model. The form of the equation depends on law governing the nucleation rate, and the general kinetics equation can become very complex<sup>24-26</sup>. The familiar form of the equation given by Eq.(1), called JMA equation or simply Avrami equation, can be obtained only if the nucleation rate is of the form  $Ct^{m-1}$ , where  $C$  and  $m$  are constants. The exponent  $n$  in Eq.(1) depends on the dimensionality of growth, and in the frame work of the original theory,  $n \geq 1$ . A discussion of this aspect can be found in an earlier article<sup>8</sup>. The kinetics data on the  $\alpha \rightarrow \omega$  transformation in Ti at high pressures (8-9 GPa) fit very well Eq.(1), but  $n < 1$ . This observation may be reviewed as a serious violation of the JMA theory. However, the present discussion is not affected by this, as the use of Eq.(1) in the earlier<sup>4,5</sup> and the present discussion is only for the purpose of extrapolating to higher pressures the parameters  $\tau$  and  $n$ , which should be considered purely empirical in nature.

## 6 Conclusions

(a) The  $\alpha \rightarrow \omega$  transformation in Ti under static pressures is thermally activated isothermal-isobaric type. (b) The characteristic time of transformation  $\tau$  decreases rapidly with increasing pressure. An extrapolation of the  $\tau$ - $p$  data beyond the highest pressure in the experiments suggests  $\tau \sim 1 \mu s$  at 19 GPa. This implies that the transformation which is thermally activated can become, at appropriate high pressure, fast enough to respond to short duration shock wave pressure pulses. (c) However, the exponent  $n$  in JMA equation vanishes at 11.4 GPa, restricting the empirical use of this equation beyond 11.4 GPa. Since Eq.(1) suggests that if  $n = 0$ , the transformation becomes time-independent, the pressure at which  $n = 0$  is identified as the pressure at which the nature of the transition changes from isothermal-isobaric to athermal-abaric. This pressure agrees with the results of the shock wave experiments which predicts a isobaric to abaric transition at  $\sim 10$  GPa.

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